X-ray photoelectron spectroscopy (XPS) report

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Job No.: 708-EST-SM-AS-2002-11-19	
Job opened by: M.T. & J.G.	**Date:2002-11-19**

Number of samples:

Sample codification: 708

708-a = OP#1 "polluted" area; exhibit lighter brown colour than "non-polluted"

708-b = OP#2 "non-polluted" area

708-c = OP#4 "slightly polluted" area

708-e = OP#7 "non-polluted" area + HF 40 % during 5 hours

708-f = OP#9 badly polymerized linseed oil covered bakelite + HF 40 % during 5 hours

708-g = OP#11 polymerized linseed oil covered bakelite + HF 40 % during 5 hours

708-h = OP#13 without linseed oil bakelite + HF 40 % during 5 hours

Nature of samples: Bakelite samples. The bakelite of samples 708-f, -g, -h used for some of the contamination experiments in the laboratory is not necessarily the same as that of samples 708-a, -b, -c, -e. The latter are cut out from a real used RPC.

Samples received: 2002-11-?

Sample storage after sample reception: air

Put under vacuum in XPS instrument: 2002-11-19

Aim of analysis:
Identify the surface contamination components and compare the different samples to determine the origin of sample 708-a (OP#1) contamination.

Results:

All the samples present a strong outgassing. Only a slice of the topmost part of the bakelite piece can be introduced in the machine and measured after pumping in the load-lock during one night.

Figure 1 presents the so-called surface concentrations as obtained by XPS for the

different samples. The sample surfaces present several contaminants, such as F, N, Na, Si, in various proportions, depending on the sample.

Polluted sample (708-a, OP#1) and slightly or non-polluted sample (708-c, OP#4 – 708-b, OP#2)

The three samples present the same contaminants (F, N, Na, Si) but the F proportion is much higher in the polluted area (6.6 at. %, OP#1) than in the others cases (~ 1.3 at. %, OP#2, OP#4), likely implying a HF etching of the damaged surface.

Polluted sample (708-a, OP#1) and non-polluted sample further exposed to HF 40% (708-e, OP#7)

The two samples are very similar, the contaminant proportions being almost identical (see Fig. 1). This is indicates that the polluted area (OP#1) has been etched by HF vapour produced during the experiment. The damages caused by the etching are close to a high HF concentration solution etching.

Polymerized linseed oil covered sample exposed to HF 40 % (708-g, OP#11)

In this case, the same contaminants are observed, but in much higher proportion than in the case of non-polluted experimental bakelite plates further exposed to HF (see Fig. 1). The reason of the strong effect of the etching solution is not explained, but the origin could be that the bakelite plates OP#9, OP#11 and OP#13 are not necessarily from the same type as those used in the OPERA experience (OP#1, OP#2, OP#4, OP#7).

Uncovered bakelite sample exposed to HF 40 % (708-h, OP#13)

As in the previous case, the contaminants are present in very high proportion on the sample surface. The N concentration is particularly important. A part of it is attributed to the bakelite glue or to melamine that often covers bakelite, as revealed by the presence of a - high binding energy (288 eV) – contribution in the C XPS line arising from C-N bonding.

Badly-polymerized linseed oil covered sample exposed to HF 40 % (708-g, OP#9)

In this case, a very low proportion of contaminants is detected on the sample surface. This probably arises from a bad wetting between the etching solution and the sample surface still covered with oil. In this case the etching solution was not efficient on the sample surface.

Notice that in the cases of the four samples etched with the HF 40 % solution, a high bonding energy (~ 403 eV) contribution appears in the N1s line that could correspond to nitrates possibly present as impurity in the HF solution.

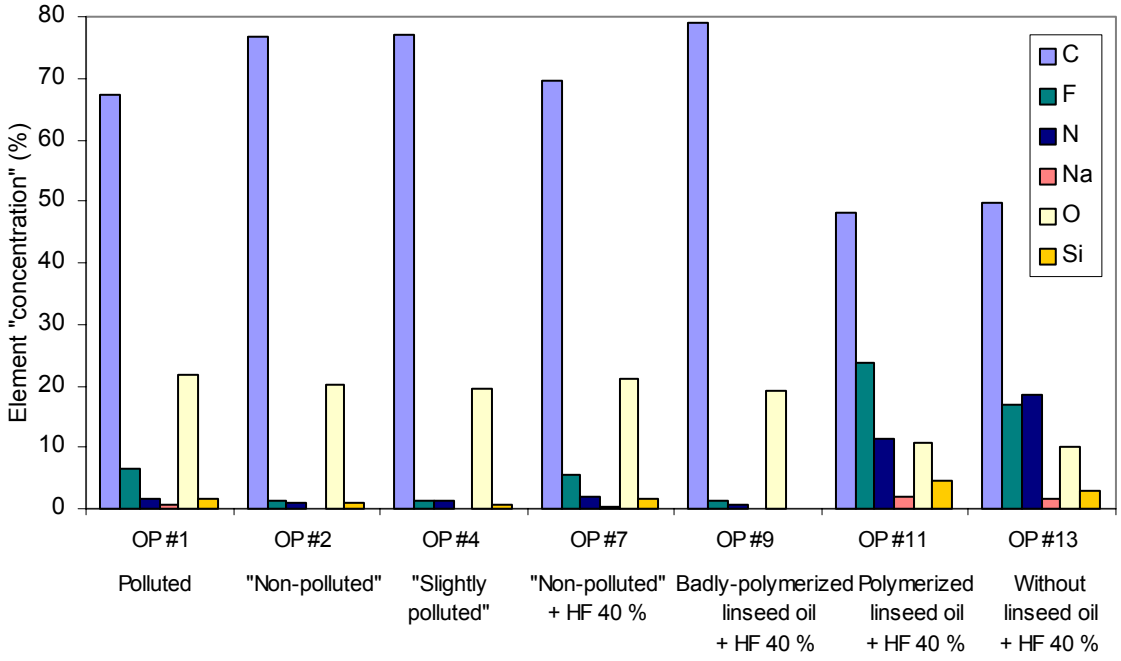

Fig. 1: Concentration of the different elements present in the surface of the samples.